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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Günter HOLZNER et al.

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Application No: 10/613,668

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Atty. Docket No.: 81455-5560

For: PERFUMING OR FLAVORING MICROCAPSULES COMPRISING A FIREPROOFING AGENT

SUBMISSION OF CERTIFIED PRIORITY DOCUMENT

Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

Sir:

Applicants have claimed priority under 35 U.S.C. § 119 of PCT Application No. PCT/IB01/02210 filed November 22, 2001. In support of this claim, a certified copy of said application is submitted herewith.

No fee or certification is believed to be due for this submission. Should any fees be required, however, please charge such fees to Winston & Strawn LLP Deposit Account No. 50-1814.

Respectfully submitted,

Date: 10/31/03

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Enclosures

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EXPRESS MAIL LIST

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The following items listed below are being filed herewith with the USPTO on **October 31, 2003**

Express Mail No. **EV 346 809 954US**		
Attorney Docket No.	Appln. Serial No.	Items - Documents filed on **October 31, 2003**
81455-5560	**10/613,668**	**Submission of Priority Document App. No. PCT/IB01/02210**

Please acknowledge receipt of these items as received by returning the enclosed postcards with the date of receipt of October 31, 2003

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PATENT COOPERATION TREATY (PCT)
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International Bureau of the World Intellectual Property Organization (WIPO)

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PCT REQUEST

5560-PCT

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0-4-1	Prepared using	PCT-EASY Version 2.92 (updated 01.03.2001)
0-5	Petition The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty	
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I	Title of Invention	PERFUMING OR FLAVOURING MICROCAPSULES COMPRISING A FIREPROOFING AGENT
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PCT REQUEST 5560-PCT

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V	**Designation of States**	
V-1	Regional Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AP: GH GM KE LS MW MZ SD SL SZ TZ UG ZW and any other State which is a Contracting State of the Harare Protocol and of the PCT EA: AM AZ BY KG KZ MD RU TJ TM and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT EP: AT BE CH&LI CY DE DK ES FI FR GB GR IE IT LU MC NL PT SE TR and any other State which is a Contracting State of the European Patent Convention and of the PCT OA: BF BJ CF CG CI CM GA GN GQ GW ML MR NE SN TD TG and any other State which is a member State of OAPI and a Contracting State of the PCT
V-2	National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned)	AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH&LI CN CO CR CU CZ DE DK DM DZ EC EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NO NZ PH PL PT RO RU SD SE SG SI SK SL TJ TM TR TT TZ UA UG US UZ VN YU ZA ZW

V-5	**Precautionary Designation Statement** In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation(s) of the State(s) indicated under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit.		
V-6	**Exclusion(s) from precautionary designations**	NONE	
VI	**Priority claim**	NONE	
VII-1	**International Searching Authority Chosen**	European Patent Office (EPO) (ISA/EP)	
VIII	**Declarations**	Number of declarations	
VIII-1	Declaration as to the identity of the inventor	-	
VIII-2	Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent	-	
VIII-3	Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application	-	
VIII-4	Declaration of inventorship (only for the purposes of the designation of the United States of America)	-	
VIII-5	Declaration as to non-prejudicial disclosures or exceptions to lack of novelty	-	
IX	**Check list**	number of sheets	electronic file(s) attached
IX-1	Request (including declaration sheets)	4	-
IX-2	Description	10	-
IX-3	Claims	2	-
IX-4	Abstract	1	EZABST00.TXT
IX-5	Drawings	0	-
IX-7	TOTAL	17	
	Accompanying Items	paper document(s) attached	electronic file(s) attached
IX-8	Fee calculation sheet	✓	-
IX-17	PCT-EASY diskette	-	Diskette
IX-19	**Figure of the drawings which should accompany the abstract**		
IX-20	**Language of filing of the international application**	English	
X-1	**Signature of applicant, agent or common representative**		
X-1-1	Name (LAST, First)	SALVATERRA-GARCIA, Maria de Lurdes	

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11-1	Date of receipt of the record copy by the International Bureau	

Perfuming or flavouring microcapsules comprising a fireproofing agent

Technical Field

The present invention relates to the field of perfumery and to the flavour industry. It concerns more particularly perfuming or flavouring microcapsules characterised by the fact that, when subjected to a sufficiently powerful ignition source, their rapid combustion reaction is weak or moderate. In fact, the microcapsules of the invention comprise an effective amount of a fireproofing agent susceptible of reducing the violence of their explosion, so as to be susceptible of being classified in a dust hazard class St-1.

Background of the Invention

Microcapsules are employed to a large extent in the perfumery and flavouring industries. They constitute delivery systems for perfuming or flavouring ingredients and can be advantageously used in a very large number of applications. The encapsulation of active substances such as perfuming or flavouring ingredients provides at the same time a protection of the ingredients there-encapsulated against "aggressions" such as oxidation or moisture and allows, on the other hand, a certain control of the kinetics of flavour or fragrance release to induce sensory effects through sequential release.

Now, the numerous advantageous properties of microcapsules in these fields are opposed by other properties that must be taken into account during their preparation, transportation, storage and handling. In fact, such delivery systems, due to their nature, and in particular to the fact that they encapsulate volatile and flammable substances, constitute combustible dusts which can, when dispersed in air or another oxygen-containing gas, form readily ignitable mixtures. When ignited by a sufficient powerful ignition source, the result is a rapid combustion reaction with advancing pressure and flame front.

These issues become important during the preparation of microcapsules. In particular, spray-drying and fluidised-bed encapsulation processes are highly concerned by this issue, as they are both based on an equipment wherein particles are suspended in hot air as fine particles and can therefore undergo explosion during their preparation.

Spray-drying is the most common encapsulation technique used to stabilise volatile substances such as flavours or fragrances, by encapsulating them in a solid form, suited to many applications. Spray-dried powders are commonly made in usual spray-drying equipment. Spray-drying is usually effected by means of a rotating disc or of multicomponent nozzles. Detailed techniques are described for instance in K. Masters, Spray-drying Handbook, Longman Scientific and Technical, 1991.

Fluidised beds are used for spraying a coating on a core material fluidised in a bed. This encapsulation technique is also well known and is described for instance in EP 70719 or in US 6,056,949, the contents of which are hereby included by reference.

Both the above-described encapsulation equipments being susceptible to explosions of particles suspended in the air, they thus have to be adapted as a function of the technical safety parameters characterising the particles there-treated. In particular, they have to be dimensioned as a function of the violence of explosions that can occur during the preparation of microcapsules. Therefore, the problem of reducing the violence of possible explosions of powder products resulting from such encapsulation processes is of paramount importance for the industry.

For the safe handling of combustible substances, it is imperative to know the dangerous properties of a product. The reliable way to characterise the combustible and explosive properties of a product is to subject a sample of the product to various tests and classify the results in accordance with the technical safety characteristics. The international standards (VDI Guideline 2263 part 1 : Dust Fires and Dust Explosions, Hazard Assessment – Protective Measures, Test Methods for the Determination of Safety Characteristics of Dusts, Beuth, Berlin, May 1990) describe the test equipments (Modified Hartmann apparatus and Close apparatus) and methods. These methods allow to determine physical constants such as the maximum explosion behaviour of a combustible dust in a closed system. A pyrotechnic igniter with a total energy of 10 kJ is used as ignition source. From test methods described in the mentioned guidelines, a characteristic constant, K_{st}, which is dust specific is determined. As there are so many such dusts produced and processed in industrial practice, for example for pharmaceutical and cereal, flour products, it is appropriate to assign this maximum explosion constant to one of the several dust explosion classes and to use these as a basis for the dimensioning of constructional protective measures. The correspondence between these classes hereafter referred as dust hazard classes, and the constant K_{st} is the following :

Dust Hazard Class	Product Specific Constant K_{St} [bar.m.s^{-1}]
St-1	> 0 to 200
St-2	< 200 to 300
St-3	< 300

Now, despite that some perfuming and flavouring ingredients are classified in a dust hazard class St-1, a large number of these ingredients and thus the microcapsules encapsulating them, and depending on the volatility of the perfuming or flavouring ingredients, are still classified under an St-2 dust hazard class and thus require production equipments specifically adapted to the violence of possible explosions, which of course can be very costly.

While solutions have been proposed for solving similar problems in other technical fields such as for instance for polymeric organic compositions which demonstrate a tendency to degrade, the perfuming and flavouring industry was never provided with an efficient solution, adapted to these products and which would solve the economic problem related to the costly equipment required to prepare St-2 classified microcapsules.

Description of the Invention

Now, we have been able to establish that fireproofing agents could be added directly to perfuming and flavouring microcapsules in an amount effective to reduce the violence of possible explosions during their preparation, when suspended in hot air. One object of the invention is thus to provide perfuming or flavouring microcapsules comprising at least one perfuming or flavouring ingredient dispersed in or adsorbed within a polymeric carrier material, characterised by the fact that the microcapsules also comprise a fireproofing agent susceptible of reducing the dust hazard explosive class of the microcapsules to St-1. It is also an object of the invention to provide methods of making and using such microcapsules.

The perfuming or flavouring microcapsules of the invention comprise an effective amount of a fireproofing agent which is capable of reducing the violence of the explosion of the microcapsules possibly induced by their suspension in the air during their preparation. This is very advantageous considering that such delivery systems are mainly

composed of highly volatile ingredients which constitute therefore combustible dusts. The invention provides an advantageous solution as regards the problem of the preparation of perfuming and flavouring microcapsules and powder products, in particular for preparations via processes involving a spray-drier or a fluidised bed, wherein fine particles are suspended in the air and are therefore more susceptible of exploding. Under an St-1 class, the violence of the explosion will be a weak or at least moderate reaction, whereas it would be a strong reaction for a dust hazard class St-2, and a very strong reaction for a dust hazard class St-3. As a consequence, the equipment used for the preparation of microcapsules according to the present invention can be dimensioned accordingly i.e. as St-1 and thus become less costly, whilst guarantying the same or better manufacture safety conditions.

Furthermore, the microcapsules and powder products of the invention not only present an advantage as regards the violence of any possible such reaction induced during their preparation, but also have proved to be less sensitive to ignition, i.e. to present a reduced tendency to explode. This characteristic may be measured and is expressed through the minimum ignition energy or MIE parameter. The MIE of a dust is defined as the lowest quantity of electrical energy stored in a capacitor which, when discharged over a spark gap, is just not sufficient to ignite the most readily ignitable dust or air mixture in a series of twenty consecutive tests, at atmospheric pressure, ambient temperature and lowest turbulence possible. The international standards (VDI Progress Report 134) state that dusts with a minimum ignition energy between 10 and 100 mJ are generally regarded as having normal ignibility, whereas, in the case of dusts with a minimum ignition energy below 10 mJ, particular attention must be paid to eliminate all sources of ignition, even weak ignition sources such as mechanical sparks or discharges of static electricity.

As regards the industry here-concerned, it turns out that some perfuming and flavouring powders possess, due to the nature of perfuming and flavouring ingredients, MIE values in the range of 1 to 10 mJ. Now, it turned out that, in a totally unexpected manner and as shown in the example below, the presence of a fireproofing agent in the composition of the microcapsules of the invention resulted in an increase in the MIE characterising values of these products, which thus reached a value above 10 mJ. This is an unexpected advantage of the invention which is moreover of paramount importance, as the microcapsules of the invention, besides their facilitated process of preparation, now present also numerous advantages as regards the requirements for their storage or even their transport, and further handling.

The fireproofing agent of the invention is preferably selected from the group consisting of sodium silicate, potassium silicate, sodium carbonate, sodium hydrogencarbonate, monoammonium phosphate or carbonate, diammonium phosphate, melamine cyanurate and chlorinated hydrocarbons. Examples of commercial products of this kind include Monnex® (origin : SICLI Matériel Incendie SA, Geneva, Switzerland), Bi-Ex® (origin : SICLI Matériel Incendie SA, Geneva, Switzerland), ABC-E® (origin : SICLI Matériel Incendie SA, Geneva, Switzerland), Tropolar® (origin : SICLI Matériel Incendie SA, Geneva, Switzerland) and ATO-33® (origin : SICLI Matériel Incendie SA, Geneva, Switzerland).

The fireproofing agent is usually present in a proportion representing from 5 to 90% by weight of the total weight of the microcapsule. Preferably, it will represent from 5 to 15% by weight relative to the total weight of the microcapsule.

The microcapsule of the invention is based on the presence of at least one perfuming or flavouring material and a polymeric carrier material.

The perfuming or flavouring ingredient, in the form of one sole ingredient or in the form of a composition, represents from 1 to 80%, and preferably from 1 to 50% by weight relative to the total weight of the microcapsule. The terms fragrance or flavour ingredient or composition as used herein are deemed to define a variety of fragrance and flavour materials of both natural and synthetic origins. They include single compounds and mixtures. Specific examples of such components may be found in the current literature, e.g. in Perfume and Flavour Chemicals by S. Arctander, Montclair, N.J. (USA) ; Fenaroli's Handbook of Flavour Ingredients, CRC Press or Synthetic Food Adjuncts by M.B. Jacobs, van Nostrand Co. Inc., and other similar text books ; and are well-known to the person skilled in the art of perfuming, flavouring and/or aromatising consumer products, i.e. for imparting an odour or a taste to a consumer product.

In one embodiment of the invention, the fragrance or flavour ingredient or composition is dispersed in a polymeric carrier material. Non limiting examples of the latter include polyvinyl acetate, polyvinyl alcohol, dextrines, natural or modified starch, vegetable gums, pectins, xanthanes, alginates, carragenans or yet cellulose derivatives such as for example carboxymethyl cellulose, methylcellulose or hydroxyethylcellulose, and generally all materials currently used for encapsulation of volatile substances.

In another embodiment, the fragrance or flavour ingredient or composition is adsorbed within a polymeric carrier material. As non limiting examples of the latter, one

can cite amorphous silica, precipitated silica, fumed silica and aluminosilicates such as zeolite and alumina.

A second object of the invention is a method for the preparation of perfuming and flavouring microcapsules, wherein a fireproofing agent is added to the microcapsules. There are several alternatives to the method for the preparation of the microcapsules of the invention. In a first embodiment, the fireproofing agent is added to an aqueous emulsion consisting of the perfuming or flavouring ingredient or composition dispersed in the polymeric carrier material. The obtained emulsion is then spray-dried in order to form a powder. Optionally, an emulsifier may be added to the initial emulsion. The encapsulation technique does not require a more detailed description herein, as it relies on conventional spray-drying techniques, which are perfectly well documented in the prior art [see for example Spray-Drying Handbook, 3rd ed., K. Masters ; John Wiley (1979)] and currently applied in the food industry or in the flavour and perfume industry.

In another embodiment, the fireproofing agent, in the form of a solid powder, is simply blended with a spray-dried powder formed from the aqueous emulsion of perfuming or flavouring ingredient or composition in the polymeric carrier material and the emulsifier.

A third alternative for the preparation of the microcapsules of the invention presenting an explosion reaction of reduced violence, is to firstly adsorb the perfuming or flavouring ingredient or composition within a porous polymeric carrier material as described above and to further coat the resulting system with a fireproofing agent. This method of preparation can be carried out in a fluidised bed apparatus, according to conventional techniques such as those described for instance in EP 70719 or in US 6,056,949 the contents of which are hereby included by reference. The particles formed by adsorption of a fragrance or flavour ingredient or composition within the carrier can thus be coated after granulation, e.g. by spraying a solution, emulsion or melt of the fireproofing agent, which forms a protective film around the core.

During the granulation process, there can also be used usual additives such as artificial sweeteners, food dyes, vitamins, antioxidants, anti-foam agents, carbonic acid generators, or additional flavorants etc. which can be added to the core material or to the spray emulsion.

The microcapsules of the invention have an average diameter varying from usually 5 to 500 μm.

The microcapsules of the invention can advantageously be used to impart, improve, enhance or modify the organoleptic properties of a great variety of edible or perfumed end products. In the field of perfumery, the perfuming microcapsules resulting from any embodiment of the process according to the invention can be incorporated in a perfuming composition added to functional products such as detergents or fabric softeners, soaps, bath or shower gels, deodorants, body lotions, shampoos and other hair-care products, household cleansers, cleaning and deodorising blocks for toilet tanks. On the other hand, in the case of flavours encapsulated, the consumer products susceptible of being flavoured by the microcapsules of the invention may include foods, beverages, pharmaceuticals and the like.

The concentrations in which the microcapsules of the invention can be incorporated in such consumer products vary in a wide range of values, which are dependent on the nature of the product to be perfumed or flavoured. Typical concentrations, to be taken strictly by way of example, are comprised in a range of values as wide as from a few ppm up to 5 or 10% of the weight of the flavouring or perfuming composition or finished consumer product into which they are included.

The invention will be now illustrated but not limited by way of the following examples wherein temperatures are given in degrees centigrade and abbreviations have the meaning common in the art.

Embodiments of the Invention

Example 1

Dry blending of a spray-dried perfuming powder and a fireproofing agent

An emulsion of the following composition was spray-dried in a spray-drier Büchi (origin : Switzerland) :

Ingredients	grams
Water	150.0
Capsul® [1)]	67.0
Perfume concentrate [2)]	33.0
Total	250.0

1) dextrin dioctenylsuccinate ; origin : National Starch, USA
2) origin : Firmenich SA, Geneva, Switzerland

The theoretical yield after evaporation of water was of 100 g of powder containing 33% of perfume.

The explosive character of the powder was measured with a Hartmann apparatus (see VDI Guideline 2263 part 1 : Dust Fires and Dust Explosions, Hazard Assessment – Protective Measures, Test Methods for the Determination of Safety Characteristics of Dusts, Beuth, Berlin, May 1990), and the powder was attributed a dust hazard class St-2.

The same powder was then mixed with diammonium phosphate in powder, in a ratio of 80:20.

The analysis of the explosive character of the homogeneous mixture, made under the same condition demonstrated that the mixture could be classified as St-1.

Example 2

Coating of combustive perfuming microcapsules with sodium silicate

Silica dioxide spheres (Tixosil 68 ; origin : Rhodia, France) were filled with the perfuming ingredient described in Example 1 and further coated with sodium silicate as follows :

Adsorption of perfume in silica dioxide

Thanks to its porous character, the silica dioxide adsorbed 60% of perfume and still stayed as a free-flowing granule without external liquid.

The explosive analysis classified the mixture as St-2.

The latter was then coated into a Kugelcoater (origin : Hüttlin, Germany) with sodium silicate, according to the following formula :

Ingredients	grams
Tioxil 68 and perfume	900
Aqueous sodium silicate solution 35%	300
Total	1200

After evaporation of water during the coating in the Kugelcoater, there were obtained about 1000 g of coated spheres coated with a sodium silicate layer.

The explosive analysis in a Hartmann apparatus classified the product as St-1. This result is a clear demonstration of the action of the sodium silicate protective layer.

Example 3

Spray-drying of an emulsion comprising a fireproofing agent

Two perfuming microencapsulated compositions were prepared by means of the following ingredients (parts by weight) :

Ingredients	Formula A	Formula B
Lavender perfume [1)]	33.0	33.0
Tween® 20 [2)]	0.3	0.3
Water	150.0	150.0
Citric acid	0.3	0.3
Capsul® [3)]	51.4	66.4
Budit® 315 [4)]	5.0	-
Monoammonium phosphate	10.0	-
Total	250.0	250.0

1) origin : Firmenich SA, Geneva, Switzerland
2) polyoxyethylene monolaurate ; origin : ICI Chemicals, Great Britain
3) dextrin dioctenylsuccinate ; origin : National Starch, USA
4) melamine cyanurate ; origin : Budenheim, Germany

The ingredients above-cited were homogenised by means of a Silverson type fast stirrer. The mixture was then spray-dried in a Sodeva apparatus with an emulsion output of 2 kg/h, drying air : 320 m^3/h at 350°C and 0.45×10^5 Pa.

There was thus obtained a fine powder, the diameter of the particles being comprised between 10 and 300 µm and the content of liquid perfume being 33% by weight.

The measure of the dust hazard class of the 2 kinds of powders showed that Formula A was classified as St-1, while Formula B was classified as St-2.

Furthermore, the minimal ignition energy (MIE) was measured for both powders (for method used, see VDI Guideline 2263 part 1 : Dust Fires and Dust Explosions, Hazard Assessment – Protective Measures, Test Methods for the Determination of Safety Characteristics of Dusts, Beuth, Berlin, May 1990). Formula A had a MIE comprised between 10 and 25 mJ, while Formula B had an MIE comprised between 5 and 10 mJ. The latter was thus considered as very reactive (very low values for its MIE) and thus should be treated as a flammable gas (such as propane or butane). On the other hand, Formula A which possessed higher values for its MIE, would thus not be ignited by electric discharges.

Claims

1. Perfuming or flavouring microcapsule comprising at least one perfuming or flavouring ingredient dispersed in or adsorbed within a polymeric carrier material, characterised in that the microcapsule further comprises an effective amount of a fireproofing agent susceptible of reducing the dust hazard explosive class of the microcapsule to St-1.

2. Perfuming or flavouring microcapsule according to claim 1, characterised in that the fireproofing agent is selected from the group consisting of sodium silicate, potassium silicate, sodium carbonate, sodium hydrogencarbonate, monoammonium phosphate or carbonate, diammonium phosphate, melamine cyanurate and chlorinated hydrocarbons.

3. Perfuming or flavouring microcapsule according to claim 1, characterised in that it comprises from 5 to 90% by weight of fireproofing agent relative to the dry weight of the microcapsule.

4. Perfuming or flavouring microcapsule according to claim 3, characterised in that it comprises from 5 to 15% by weight of fireproofing agent relative to the dry weight of the microcapsule.

5. Perfuming microcapsule according to claim 1, characterised in that it comprises up to 80% by weight of perfume relative to the total weight of the microcapsule.

6. Perfuming microcapsule according to claim 1, characterised in that it comprises up to 50% by weight of perfume relative to the total weight of the microcapsule.

7. Method for the preparation of perfuming or flavouring microcapsules as defined in claim 1, which comprises adding a fireproofing agent to an aqueous emulsion of the perfuming or flavouring ingredient in the carrier polymeric material, and spray-drying the obtained emulsion to form a powder.

8. Method for the preparation of perfuming or flavouring microcapsules as defined in claim 1, which comprises impregnating a porous polymeric carrier material with the perfuming or flavouring ingredient and coating the resulting system with the fireproofing agent.

9. Method for the preparation of perfuming or flavouring microcapsules as defined in claim 1, which comprises spray-drying an aqueous emulsion of the perfuming or flavouring ingredient in the polymeric carrier and dry blending the powder obtained with the fireproofing agent also in the form of a powder.

10. Use of a fireproofing agent in the composition of a perfuming or flavouring microcapsule for reducing the violence of the explosion of the microcapsule during its suspension in the air.

11. Use according to claim 10, characterised in that the fireproofing agent is selected from the group consisting of sodium silicate, potassium silicate, sodium carbonate, sodium hydrogencarbonate, monoammonium phosphate or carbonate, diammonium phosphate, melamine cyanurate and chlorinated hydrocarbons.

12. A perfumed product selected from the group consisting of a detergent, a fabric softener, a soap, a bath or shower gel, a deodorant, a body lotion, a shampoo or another hair-care product, a household cleaner and a cleaning and deodorising block for toilet tanks, characterised in that it comprises perfuming microcapsules according to any one of claims 1 to 6.

13. A food, beverage or pharmaceutical product, characterised in that it comprises flavouring microcapsules according to any one of claims 1 to 6.

Abstract

Perfuming or flavouring microcapsules comprising, further to a perfuming or flavouring ingredient and a carrier material, a fireproofing agent, proved to undergo, when suspended in hot air during their preparation, an explosion of a reduced violence.